

THAT WHICH IS CLAIMED IS:

1. A method of producing a semi-insulating silicon carbide crystal with a controlled nitrogen content, the method comprising:

introducing an ambient gas containing hydrogen into a sublimation growth  
5 chamber;

heating a silicon carbide source powder to sublimation in the hydrogen ambient growth chamber while,

heating and then maintaining a silicon carbide seed crystal in the hydrogen ambient growth chamber to a second temperature below the temperature of the source  
10 powder, at which second temperature sublimed species from the source powder will condense upon the seed crystal,

continuing to heat the silicon carbide source powder until a desired amount of silicon carbide crystal growth has occurred upon the seed crystal;

while maintaining an ambient concentration of hydrogen in the growth  
15 chamber sufficient to minimize the amount of nitrogen incorporated into the growing silicon carbide crystal; and

heating the crystal to increase the number of point defects in the crystal to an amount that renders the resulting silicon carbide crystal semi-insulating.

20        2. A method according to Claim 1 wherein the step of heating the crystal to increase the number of point defects comprises maintaining the source powder and the seed crystal during sublimation growth at respective temperatures high enough to increase the number of point defects in the growing crystal to an amount that renders the resulting silicon carbide crystal semi-insulating.

3. A method according to Claim 1 wherein the step of heating the crystal to increase  
the number of point defects comprises heating a silicon carbide crystal to a temperature  
above the temperatures required for CVD growth of silicon carbide from source gases, but  
less than the temperatures at which disadvantageously high rates of silicon carbide  
5 sublimation occur under the ambient conditions to thereby thermodynamically increase the  
concentration of point defects and resulting states in the crystal; and then cooling the heated  
crystal to approach room temperature at a sufficiently rapid rate to minimize the time spent in  
the temperature range in which the defects are sufficiently mobile to disappear or be re-  
annealed into the crystal to thereby produce a silicon carbide crystal with a concentration of  
10 point defect states that is greater than the concentration of point defect states in an otherwise  
identically grown silicon carbide crystal that has not been heated and cooled in this manner.

4. A method according to Claim 1 comprising introducing the ambient  
hydrogen into the growth chamber at a pressure between about 0.1 and 50 Torr.

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5. A method according to Claim 1 comprising introducing the ambient  
hydrogen into the growth chamber at a flow rate of between about 10 and 1000  
standard cubic centimeters per minute.

20        6. A method according to Claim 1 comprising heating a seed crystal having a  
polytype selected from the group consisting of 3C, 4H, 6H, and 15R polytype of  
silicon carbide.

25        7. A method according to Claim 1 comprising maintaining the silicon carbide  
source powder at a temperature of between about 2000°C and 2500°C and  
maintaining the seed crystal at a temperature that is between about 50°C and 350°C  
lower than the temperature of the source powder.

8. A method according to Claim 1 comprising heating a silicon carbide source powder in which the amounts of deep level trapping elements in the source powder are below the levels that can be detected by secondary ion mass spectroscopy (SIMS).

5       9. A method according to Claim 1 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $2 \times 10^{15}$  nitrogen atoms per cubic centimeter.

10     10. A method according to Claim 1 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $1 \times 10^{15}$  nitrogen atoms per cubic centimeter.

11. A method according to Claim 1 comprising introducing a hydrocarbon species into the growth chamber to establish the hydrogen ambient.

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12. A semi-insulating silicon carbide crystal produced by the method of Claim 1 having a concentration of nitrogen atoms less than about  $2 \times 10^{15} \text{ cm}^{-3}$ .

13. A semi-insulating silicon carbide crystal produced by the method of Claim 1 having a concentration of nitrogen atoms less than about  $1 \times 10^{15} \text{ cm}^{-3}$ .

14. A semi-insulating silicon carbide crystal produced by the method of Claim 1 having a resistivity of at least  $1 \times 10^5 \text{ ohm-cm}$ .

15. A method of producing a semi-insulating silicon carbide crystal with a controlled nitrogen content, the method comprising:

introducing an ambient gas containing hydrogen into a sublimation growth chamber;

5 heating a silicon carbide source powder to sublimation in the hydrogen ambient growth chamber while,

heating and then maintaining a silicon carbide seed crystal in the hydrogen ambient growth chamber to a second temperature below the temperature of the source powder, at which second temperature sublimed species from the source powder will  
10 condense upon the seed crystal,

continuing to heat the silicon carbide source powder until a desired amount of silicon carbide crystal growth has occurred upon the seed crystal;

while maintaining an ambient concentration of hydrogen in the growth chamber sufficient to passivate the growing silicon carbide crystal against the

15 incorporation of nitrogen to thereby minimize the amount of nitrogen incorporated into the growing silicon carbide crystal; and

heating the crystal to increase the number of point defects in the crystal to an amount that renders the resulting silicon carbide crystal semi-insulating.

20 16. A method according to Claim 15 wherein the step of heating the crystal to increase the number of point defects comprises maintaining the source powder and the seed crystal during sublimation growth at respective temperatures high enough to increase the number of point defects in the growing crystal to an amount that renders the resulting silicon carbide crystal semi-insulating.

17. A method according to Claim 15 wherein the step of heating the crystal to increase the number of point defects comprises heating a silicon carbide crystal to a temperature above the temperatures required for CVD growth of silicon carbide from source gases, but less than the temperatures at which disadvantageously high rates of silicon carbide 5 sublimation occur under the ambient conditions to thereby thermodynamically increase the concentration of point defects and resulting states in the crystal; and then cooling the heated crystal to approach room temperature at a sufficiently rapid rate to minimize the time spent in the temperature range in which the defects are sufficiently mobile to disappear or be re-annealed into the crystal to thereby produce a silicon carbide crystal with a concentration of 10 point defect states that is greater than the concentration of point defect states in an otherwise identically grown silicon carbide crystal that has not been heated and cooled in this manner.

18. A method according to Claim 15 comprising introducing the ambient hydrogen into the growth chamber at a pressure of between about 0.1 and 50 Torr.

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19. A method according to Claim 15 comprising introducing the ambient hydrogen into the growth chamber at a flow rate of between about 10 and 1000 standard cubic centimeters per minute.

20 20. A method according to Claim 15 comprising maintaining the silicon carbide source powder at a temperature of between about 2000°C and 2500°C and maintaining the seed crystal at a temperature that is between about 50°C and 350°C lower than the temperature of the source powder.

25 21. A method according to Claim 15 comprising heating a seed crystal having a polytype selected from the group consisting of 3C, 4H, 6H, and 15R polytype of silicon carbide.

22. A method according to Claim 15 comprising heating a silicon carbide source powder in which the amounts of deep level trapping elements in the source powder are below the levels that can be detected by secondary ion mass spectroscopy (SIMS).

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23. A method according to Claim 15 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $2 \times 10^{15}$  nitrogen atoms per cubic centimeter.

10        24. A method according to Claim 15 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $1 \times 10^{15}$  nitrogen atoms per cubic centimeter.

15        25. A semi-insulating silicon carbide crystal produced by the method of Claim 13 having a concentration of nitrogen atoms less than about  $2 \times 10^{15} \text{ cm}^{-3}$ .

26. A semi-insulating silicon carbide crystal produced by the method of Claim 15 having a concentration of nitrogen atoms less than about  $1 \times 10^{15} \text{ cm}^{-3}$ .

20        27. A semi-insulating silicon carbide crystal produced by the method of Claim 15 having a resistivity of at least  $1 \times 10^5 \text{ ohm-cm}$ .

28. A method of producing semi-insulating silicon carbide crystal with a controlled nitrogen content, the method comprising:

heating and maintaining a silicon carbide source powder to sublimation while,

heating and maintaining a silicon carbide seed crystal to a temperature below  
5 the temperature of the source powder, at which temperature sublimed species from  
the source powder condense upon the seed crystal to form a continuously expanding  
growth surface of silicon carbide crystal;

passivating the silicon carbide growth surface with hydrogen atoms to reduce  
the incorporation of nitrogen from the ambient atmosphere into a resulting silicon  
10 carbide crystal,

heating the crystal to increase the number of point defects in the crystal to an  
amount that renders the resulting silicon carbide crystal semi-insulating.

29. A method according to Claim 28 wherein the step of heating the crystal to  
15 increase the number of point defects comprises maintaining the source powder and  
the seed crystal during sublimation growth at respective temperatures high enough to  
increase the number of point defects in the growing crystal to an amount that renders  
the resulting silicon carbide crystal semi-insulating.

20       30. A method according to Claim 28 wherein the step of heating the crystal to  
increase the number of point defects comprises heating a silicon carbide crystal to a  
temperature above the temperatures required for CVD growth of silicon carbide from source  
gases, but less than the temperatures at which disadvantageously high rates of silicon carbide  
sublimation occur under the ambient conditions to thereby thermodynamically increase the  
25 concentration of point defects and resulting states in the crystal; and then cooling the heated  
crystal to approach room temperature at a sufficiently rapid rate to minimize the time spent in  
the temperature range in which the defects are sufficiently mobile to disappear or be re-

annealed into the crystal to thereby produce a silicon carbide crystal with a concentration of point defect states that is greater than the concentration of point defect states in an otherwise identically grown silicon carbide crystal that has not been heated and cooled in this manner.

5        31. A method according to Claim 28 comprising passivating the growth surface with hydrogen atoms by heating the source crystal and the seed crystal in a hydrogen ambient atmosphere.

10      32. A method according to Claim 28 comprising passivating the growth surface with hydrogen atoms by adding hydrogen to the ambient atmosphere at a pressure of between about 0.1 and 50 Torr.

15      33. A method according to Claim 28 comprising passivating the growth surface with hydrogen atoms by adding hydrogen to the ambient atmosphere at a flow rate of between about 10 and 1000 standard cubic centimeters per minute.

20      34. A method according to Claim 28 comprising maintaining the silicon carbide source at a temperature of between about 2000°C and 2500°C and maintaining the seed crystal at a temperature that is between about 50°C and 350°C lower than the temperature of the source powder.

35. A method according to Claim 28 comprising heating a seed crystal having a polytype selected from the group consisting of 3C, 4H, 6H, and 15R polytype of silicon carbide.

36. A method according to Claim 28 comprising heating to sublimation a silicon carbide source powder in which the amounts of deep level trapping elements in the source powder are below the levels that can be detected by secondary ion mass spectroscopy (SIMS).

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37. A method according to Claim 28 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $2 \times 10^{15}$  nitrogen atoms per cubic centimeter.

10        38. A method according to Claim 28 comprising introducing a sufficient amount of ambient hydrogen into the growth chamber to yield a growing silicon carbide crystal with less than about  $1 \times 10^{15}$  nitrogen atoms per cubic centimeter.

15        39. A semi-insulating silicon carbide crystal produced by the method of Claim 28 having a concentration of nitrogen atoms less than about  $2 \times 10^{15} \text{ cm}^{-3}$ .

40. A semi-insulating silicon carbide crystal produced by the method of Claim 28 having a concentration of nitrogen atoms less than about  $1 \times 10^{15} \text{ cm}^{-3}$ .

20        41. A semi-insulating silicon carbide crystal produced by the method of Claim 28 having a resistivity of at least  $1 \times 10^5 \text{ ohm-cm}$ .

42. A method of producing semi-insulating silicon carbide crystal by heating and maintaining a silicon carbide source powder to sublimation in a growth chamber, while heating and maintaining a silicon carbide seed crystal in the growth chamber to a second temperature below the temperature of the source powder, at which second  
5 temperature sublimed species from the source powder condense upon the seed crystal to continuously grow a silicon carbide crystal while maintaining the source powder and the seed crystal during sublimation growth at respective temperatures high enough to increase the number of point defects in the growing crystal to an amount that renders the resulting silicon carbide crystal semi-insulating, the method  
10 comprising maintaining an ambient concentration of hydrogen in the growth chamber sufficient to minimize the amount of nitrogen incorporated into the silicon carbide crystal.